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DETERIORATION OF COMMERCIALLY PACKED CHLORINATED LIME

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PURPOSE OF INVESTIGATION

Chlorinated lime, sometimes called "bleaching powder" or improperly "chloride of lime," is extensively used as a bleaching agent and as a germicide and deodorant. Although the chemical composition of this substance has not been definitely determined, it is generally considered that the active constituent is calcium oxychloride, according to Odling's formula, $Ca\begin{smallmatrix}Cl\\OCl\end{smallmatrix}$, accompanied by calcium chloride and free lime in varying quantities. The chemistry of hypochlorites is fully discussed by Lunge (*4*).[1]

The activity of chlorinated lime as a bleaching agent or as a disinfectant is expressed in terms of the "available chlorine" content. Hooker (*3*) defines this as "the whole amount of free chlorine that becomes available in decomposing chlorinated lime with strong acid."

Despite the use of the term "available chlorine," the activity of chlorinated lime as a germicide or deodorant is commonly believed to depend not on its chlorinating but on its oxidizing power (*3*). The carbon dioxide of the air and possibly weak organic acids contained in tap water or in soil liberate free hypochlorous acid, which is held to be the active oxidizing agent of chlorinated lime. This acid gives up its oxygen very rapidly, as may be expressed by the equation $HClO=HCl+O$. Nascent oxygen so formed decomposes most organic matter with which it comes in contact.

The fact that the strength of chlorinated lime decreases with age has long been known. Since its activity as a germicide is in proportion to its available chlorine content at the time of use, this deterioration is very important to consumers, who not only suffer a monetary loss when they fail to get what they pay for but also find the product ineffective as a germicide. In the false security given

[1] Italic numbers in parentheses refer to "Literature cited," p. 19.

by its use, other precautions may be omitted, with very serious injury to health as the result.

The investigation here reported was undertaken for the purpose of determining the rate of deterioration of chlorinated lime, as expressed by its loss of available chlorine, when stored under different conditions of temperature and packing.

RESULTS OF PREVIOUS INVESTIGATIONS

Thümmel (*12*) determined the available chlorine content of chlorinated lime bought from local druggists when received and after it had been stored for two months (July and August) in open vessels in a place protected from light. He found that the loss in available chlorine ranged from 0.6 to 6.6 per cent. The loss in the upper layers of the samples was slightly greater than that in the lower portions. Table 1 shows the results of another determination of the loss of available chlorine in samples of chlorinated lime when packed in glass containers and stored under varying conditions conducted by Thümmel.

TABLE 1.—*Available chlorine in chlorinated lime lost under varying storage conditions (Thümmel)*

Storage conditions	Available chlorine	Loss
	Per cent	*Per cent*
At time of packing	35.9	
Protected from the light for 2 months:		
Open in cellar	29.7	6.2
Closed on floor	31.2	4.7
Open on floor	23.0	12.9
In sunlight for 16 months	5.9	30.0

Pattinson (*7*) made deterioration tests on chlorinated lime in three casks, each holding 6 hundredweight of the material. Twelve subsamples from each cask were stored for 11 months in bottles in a cellar where the temperature ranged from 38° to 62° F. This investigator concluded that chlorinated lime kept for about 12 months, in either flasks or bottles, at temperatures below 60° F. loses not more than 2 to 3 per cent of available chlorine and less than 1 per cent of total chlorine. Later Pattinson (*8*) made tests in which the subsamples were kept for 11 months at 60°, 70°, and 80° F. At the end of the 11 months chlorinated lime originally containing 36.1 per cent of available chlorine contained 30.1 per cent when kept at 60°, 28.3 per cent when kept at 70°, and 19.1 per cent when kept at 80°. During this period there was also a regular and corresponding increase of chlorine existing as chloride. In the samples which were kept at 60° F. the chlorate content decreased to zero; in the other samples it decreased to zero at first and then rose, the samples kept at 80° F. showing the largest increase.

Boyer and Durand (*1*) found that dry chlorinated lime undergoes a change about twice as quickly in the open air as when exposed in a closed room, and somewhat more rapidly in light than in darkness. This change takes place much more promptly in an atmosphere containing hydrogen sulphide, carbon dioxide, or hydrochloric acid vapors than in ordinary air.

Smith (*11*) stored subsamples of four lots of bleaching powder under similar conditions for one year and analyzed newly-opened

cans or bottles at intervals. One sample stored in sealed bottles showed a loss of 8.2 per cent available chlorine in 11 months and one of 10.8 per cent in 12 months. During 11 months one showed an 8.2 per cent loss when stored in sealed bottles and an 8.4 per cent loss when stored in zinc cans. Another sample, originally containing 37 per cent available chlorine, lost 12.25 per cent from February to October when stored in iron (tin-coated) cans. The cans were corroded at the end of the storage period. The fourth sample, containing originally 41.9 per cent available chlorine, lost 3.64 per cent when held in sealed bottles from April to October.

Schwarz (*10*) noticed that the results in a bleachery using about one cask of chlorinated lime a week were irregular, although the product obtained was of good quality. The trouble was traced to deterioration of the chlorinated lime when kept in open packages. The material originally testing 34.65 per cent of available chlorine lost when carefully covered and protected only 0.2 to 0.3 per cent in two weeks; the unprotected product lost 4.61 per cent.

In 1915 the laboratory of the inland revenue department of Canada (*5*) examined 101 samples of chlorinated lime, each supposed to contain 30 per cent of available chlorine. Analyzed by the method of the British Pharmacopœia (1914), 21 samples yielded 30 per cent available chlorine, 41 samples 25 per cent, 26 samples 20 per cent, 8 samples 10 per cent, and 5 samples less than 10 per cent.

Rettie, Smith, and Ritchie (*9*) state that chlorinated lime loses 96 per cent of its available chlorine in eight weeks at 37° C. (98.6° F.) and in two weeks at 45° C. (113° F.). By mixing with chlorinated lime a quantity of freshly burnt quicklime from 10 to 15 per cent in excess of the quantity calculated to be necessary to react with the water present, the keeping qualities are much improved, according to these writers.

Macculloch (*6*) tested a sample of "stabilized bleach" (chlorinated lime mixed with 20 per cent powdered quicklime) with ordinary chlorinated lime for comparison. These tests, which were made in India, gave the results shown in Table 2.

TABLE 2.—*Available chlorine lost in bleaching powder stored for varying periods (Macculloch)*

Time and place of analysis	Available chlorine	
	In ordinary bleach	In "stabilized" bleach
	Per cent	*Per cent*
In England	35.00	25.00
On reaching Madras	20.04	18.40
After reaching Madras:		
6 weeks	12.52	16.56
10 weeks	4.68	15.90
14 weeks	.42	15.05
26 weeks		12.08
42 weeks		10.00
52 weeks		8.50
104 weeks		3.48

Gizolme (*2*) found that in nine months chlorinated lime kept in wooden barrels in a covered shed lost half of its available chlorine. He concludes that the loss of available chlorine depends on the purity and the degree of desiccation of the product. Humid or impure samples show a higher rate of deterioration than dry and pure samples.

OUTLINE OF INVESTIGATION

For the purpose of the investigation here reported, samples of chlorinated lime were collected at the packers' plants. Packers buy their chlorinated lime from manufacturers in drums containing about 700 pounds and repack it into packages ranging usually from 5 ounces to 10 pounds in weight.

At each plant the contents of an unopened drum recently received was thoroughly mixed by shoveling. The bleaching powder was then repacked, either by hand or by machinery, according to the commercial method of the packer, into small containers. Available chlorine was determined on the original material at the plant at the time of packing.

The packages of each commercial size thus prepared were shipped to Washington, where a complete analysis was made of the contents of one package of each size. The other packages were stored in a warehouse. Weekly ranges of temperature were recorded. Determinations of available chlorine were made at the end of 2, 4, 6, and 8 weeks, and monthly thereafter, a newly opened package being used for the analysis in every case. At the end of the period complete analysis was made of a sample from a package of each size. Two series of tests were conducted—one on chlorinated lime made during the summer and one on that made during the winter. In all, the products of three manufacturers and five packers were tested.

In addition to the samples packed at the plants, several 10-pound samples from each manufacturer were mixed and subsampled into glass bottles, which were closed with paraffined cork stoppers. Some of these were stored in a dark closet and others in the light. At monthly intervals they were tested for their available chlorine content.

An imported sample of chlorinated lime, testing 35.9 per cent available chlorine when received (February 15, 1923), was subsampled into 5-ounce cardboard (paraffined) containers with metal tops, of the type used in commercial practice. These were tested at monthly intervals. Another imported sample, testing 34.7 per cent available chlorine, was packed into glass bottles with paraffined cork stoppers, stored in the dark, and tested monthly.

CONTAINERS USED

Realizing that the type of container used by the packer would probably be one of the factors influencing the deterioration of chlorinated lime, the investigators endeavored to obtain a representative assortment of containers of the various common types and sizes.

The chlorinated lime manufactured in the summer was packed in containers of the following types. Packer A: 12-ounce size, paraffined fiber container with lacquered metal ends; 10-pound size, lacquered metal can with crimped top. Packer B: 12-ounce size, paraffined fiber container with top glued on; 10-pound size, lacquered metal can with crimped top. Packer C: 12-ounce size, paraffined fiber container with top of same material glued on; 5-pound size, paraffined fiber container with top of same material glued on. Packer D: 5 and 12 ounce sizes, paraffined fiber container with lacquered metal ends; 5-pound size, paraffined fiber container with top of same material glued on; 10-pound size, lacquered metal can with crimped top. Packer E: 10-pound size, lacquered metal can with crimped top, with a layer of lime placed on top of the chlorinated

lime. (Packer believed that this layer of lime helped to prevent deterioration.)

The chlorinated lime manufactured in the winter was packed in containers of the following types. Packer A: 12-ounce size, paraffined fiber container with lacquered metal ends; 5 and 10 pound sizes, lacquered metal can with crimped top. Packer B: 8-ounce size, paraffined fiber container with top of same material glued on; 5 and 10 pound sizes, lacquered metal can with crimped top. Packer C: 12-ounce size, paraffined fiber container with top of same material glued on; 5-pound size, paraffined fiber container with top of same material glued on. Packer D: 5 and 12 ounce sizes, paraffined fiber container with lacquered metal ends; 5-pound size, paraffined fiber container with top of same material glued on; 10-pound size, lacquered metal can with crimped top. All samples were packed by machinery, except those from packer C, which were packed by hand, using a small scoop.

METHODS OF ANALYSIS

AVAILABLE CHLORINE

The following well-known method devised by Penot, described in most standard textbooks on analysis, was used for the determination of available chlorine.

Weigh 7.092 grams of the thoroughly mixed sample into a mortar and triturate with 30 to 40 cubic centimeters of water. Add more water and allow the insoluble residue to settle. Pour the clear solution into a liter volumetric flask, and repeat the trituration until only a silicious residue remains. Rinse the mortar and pestle, catch the wash water in the flask, dilute the solution to the mark, and mix. Immediately pipet a 50 cubic centimeter aliquot into a 200 cubic centimeter Erlenmeyer flask and titrate with 0.1 *N* sodium arsenite solution, using starch-iodide paper as an outside indicator. The number of cubic centimeters of 0.1 *N* solution consumed gives directly the percentage of available chlorine in the sample.

Prepare the sodium arsenite solution used for these determinations by dissolving in water the required theoretical quantity (4.948 grams) of resublimed arsenious oxide and 10 grams of anhydrous sodium carbonate for every liter of standard solution to be made. Standardize the solution by comparing it with a standard 0.1 *N* solution of sodium thiosulphate, titrating both the sodium arsenite and the sodium thiosulphate solutions against a known quantity of standard iodine solution. Standardize the thiosulphate solution by titrating it with 0.1 *N* potassium permanganate solution according to the method described by Treadwell and Hall (*13*). Standardize the permanganate in the usual manner against pure sodium oxalate obtained from the Bureau of Standards, United States Department of Commerce.

This roundabout method of standardizing the sodium arsenite solution was adopted in order to have it based upon a recognized Government standard.

CHLORIDE CHLORINE

The chloride chlorine was determined from the solution prepared for the determination of available chlorine by the following method described by Lunge (*4*):

After the sodium arsenite titration has been made, add a small excess of nitric acid, neutralize the solution with calcium carbonate,

and titrate with 0.1 *N* silver nitrate, using as an indicator potassium chromate or the sodium arsenate formed in the solution.

In either case a blank determination on the reagents used should be made. This titration gives the sum of the available and chloride chlorine. The percentage of chloride chlorine present is found by deducting the percentage of available chlorine from this value.

TOTAL CHLORINE

The following method, which is described by Pattinson (*8*), was used to determine the total chlorine:

Treat with sulphurous acid a 50-cubic centimeter aliquot of the solution prepared for the determination of available chlorine and heat the mixture to boiling, to reduce chlorates and expel the excess sulphurous acid. Add two or three drops of nitric acid to insure complete removal of the sulphurous acid. Cool the solution, neutralize with calcium carbonate, and titrate with silver nitrate, as outlined in the method for chloride chlorine.

This method gives total chlorine. The difference between the available chlorine plus the chloride chlorine and this result represents the chlorine existing in a higher oxidized form, probably as chlorates.

CALCIUM, MAGNESIUM, IRON, ALUMINUM, AND SILICA

The following method was used to determine the calcium, magnesium, iron, aluminum, and silica:

To 2 grams of the sample in water, in a 250-cubic centimeter beaker, add 25 cubic centimeters of strong hydrochloric acid (specific gravity 1.184) and 50 cubic centimeters of 3 per cent hydrogen peroxide solution. Evaporate this solution on a steam bath, bake on a hot plate for one-half hour to dehydrate silica, take up in dilute hydrochloric acid (1+9), and filter off the silica. Ignite the precipitate and weigh as silica (SiO_2).

Add ammonia in slight excess to the filtrate and washings, and boil the solution until the odor of ammonia is barely perceptible. Filter the combined hydroxides of iron and aluminum, and collect the filtrate and washings in a 250-cubic centimeter volumetric flask. Ignite the precipitate and weigh as iron and aluminum oxides (R_2O_3).

Dilute the filtrate to mark, mix, and precipitate the calcium in the usual manner with ammonium oxalate, using a 50-cubic centimeter aliquot and reprecipitating. Dissolve the precipitate in dilute sulphuric acid (1+3) and titrate with 0.2 *N* potassium permanganate. Report the result as calcium oxide (CaO).

Determine magnesium in the combined filtrate and washings from the calcium determination by precipitating with disodium phosphate solution, igniting, and weighing as magnesium pyrophosphate ($Mg_2P_2O_7$).

CARBON DIOXIDE

Expel carbon dioxide in an evolution flask, passing the gas through wash bottles containing potassium iodide solution, and absorbing the carbon dioxide with standard barium hydroxide solution. Titrate the excess barium hydroxide with standard hydrochloric acid. The apparatus used (fig. 1) consists of the following: An evolution flask (*A*), connected with a soda-lime tube (*B*), dropping funnel (*C*), and Kjeldahl trap (*D*), wash bottles (*E*), containing glass beads and

potassium iodide solution, and a Meyer absorption tube (*F*), containing standard 0.1 *N* barium hydroxide solution.

The following method was used:

Weigh 5 grams of the sample into the evolution flask (*A*), attach the stopper carrying the spray trap (*D*) and soda-lime guard tube (*B*), and add 50 cubic centimeters of hydrogen peroxide solution through the dropping funnel (*C*). When action ceases, add 30 cubic centimeters of dilute hydrochloric acid (1+3) and draw air slowly through the apparatus. The evolved gases are freed from chlorine by the potassium iodide wash bottles (*E*), and the carbon dioxide is absorbed in 50 cubic centimeters of standard barium hydroxide solution in the Meyer tube (*F*). Draw air through the

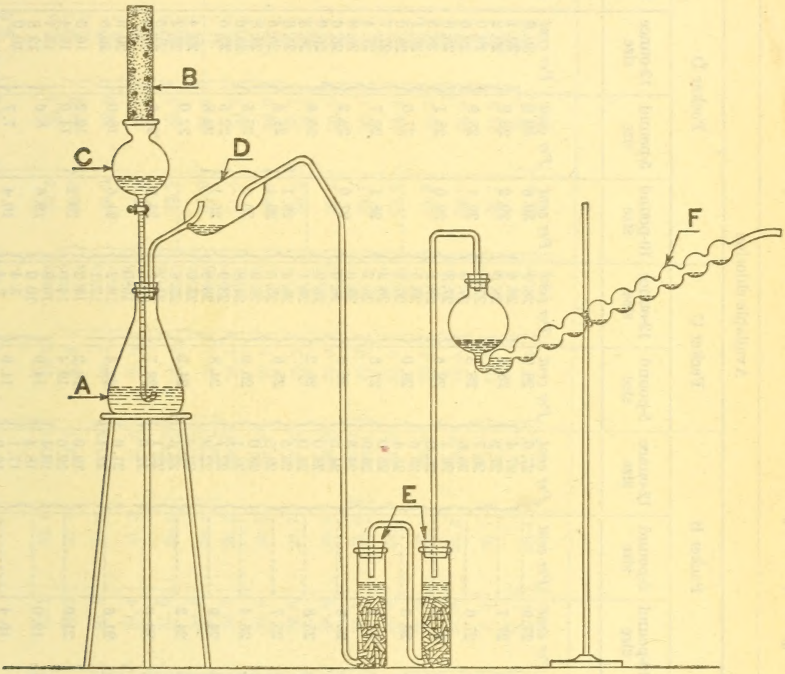

FIG. 1.—Apparatus for the determination of carbon dioxide

apparatus for 20 minutes, disconnect the Meyer tube, and pour its contents into a 500-cubic centimeter Erlenmeyer flask. Thoroughly wash the Meyer tube and add the washings to the contents of the flask. Titrate the solution without filtering off the precipitated carbonate, using 0.1 *N* hydrochloric acid and phenolphthalein as an indicator.

Repeated determinations, both with and without filtering off the barium carbonate, gave check results.

RESULTS OF INVESTIGATION

The loss of available chlorine during storage is shown in Table 3 and graphically in Figures 2, 3, 4, and 5. The results on the samples from packers C and D were not used in calculating the averages used in plotting the graph for the summer-packed material for the reasons given on page 14.

TABLE 3.—*Effect of storage on available chlorine content of chlorinated lime*

Period of storage when sampled	Available chlorine															
	Packer A			Packer B			Packer C		Packer D				Packer E	Average	Temperature range
	10-pound size	5-pound size	12-ounce size	10-pound size	5-pound size	12-ounce size	5-pound size	12-ounce size	10-pound size	5-pound size	12-ounce size	5-ounce size	10-pound size		
Summer series (packed week of Sept. 6, 1922):	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*° F.*
When packed	27.0		27.0	27.9		27.9	28.9	28.9	30.6	30.6	30.6	30.6	34.1	29.5	
1 week	27.2		26.4 26.5	27.7		27.4 27.2	28.6	28.4 28.6	29.2	28.9	29.1 29.2	29.2 29.4	34.0	28.8	61–85
2 weeks	27.1		25.9 25.9	27.6		26.7 26.6	28.3	27.8 27.9	29.1	28.9	29.0 29.0	29.2	33.8	28.5	54–76
4 weeks	26.7		25.5 25.5	27.2		26.1 26.0	28.5	27.3 27.1	29.0	28.1	28.6 28.5	28.3 28.3	33.2	28.0	51–80
6 weeks	26.8		25.3 25.2	26.5		25.6 25.4	28.0	26.5 26.3		27.6	28.1 28.0	27.6 27.5	32.5	27.4	44–75
8 weeks	26.7		25.0 25.0	26.1		25.0 24.9	27.6	26.2 26.2	28.1	26.7	27.7 27.4	27.4 27.3	32.0	27.1	46–72
3 months	26.6		24.6 24.7	25.8		24.5 24.5	26.6	26.0 26.0	27.0	26.2	26.9 26.6	26.8 26.6	31.3	26.5	44–69
4 months	26.5		24.5 24.7	25.8		24.0 24.0	26.2	25.9 26.1		25.4	26.0 26.3	26.6 26.3	30.5	26.2	48–68
5 months	25.5		24.6 24.4	25.7		23.5 23.5	25.6	25.4 25.5	26.1 26.1	24.4	25.6 25.5	25.5	30.0	25.6	49–65
6 months	24.1		24.3 24.2	25.4		23.0 23.1	25.0	24.9 24.8	25.6	23.6	24.8 24.6	24.7 24.5	29.4	25.0	32–76
7 months	23.8		24.4 24.6	24.9		22.7 22.9	24.8	23.9 23.8	25.1	22.5 22.8	23.6	23.5	28.6	24.3	34–78
8 months	23.4		23.1 23.1	24.2		22.4 22.6	24.2	23.0 22.8		22.0	22.4 22.2	22.4 22.2	27.7	23.5	36–80
9 months	23.0		22.7 22.5	23.0		22.1 22.0	23.7	21.2 20.6	23.0	20.6	21.5 21.5	21.7 21.6	25.4	22.5	50–89
10 months	22.4		21.9 21.9	21.6		21.6 21.4	22.4	16.0 18.2 17.1	21.5	16.0	20.9 20.8	20.9 20.8	24.5	21.0	72–95
11 months	21.7		21.0	21.0		19.9 19.6	18.2 19.4	14.2 14.0	20.2	12.2 14.0	16.3 16.4	17.2	23.3	18.8	72–90
12 months	20.4		19.5 19.8	19.0		18.4 18.4	14.9	10.9 10.0	19.6	8.6	13.1 12.8	14.1 14.4	22.0	16.4	68–90
13 months	19.3		18.0	15.4		17.1 16.9	11.9	3.4 7.4	10.4	7.7	10.0	8.1 8.4	21.1	13.1	52–88
14 months	18.4		16.7 17.0	18.0		16.2 15.8	11.5	5.4 3.2	19.8	6.6	8.9 8.6	7.8 7.1			40–80

15 months	18.2		15.9 16.3	15.9		16.0 15.0	10.8 9.5	1.9 4.8	18.0	7.7 6.2	7.4 7.2 6.6	6.4 6.0			42-60
Winter series (packed week of Feb. 5, 1923):															
When packed	29.3	29.3	29.3	32.1	32.1	32.1	30.3	30.3	33.5	33.5	33.5	33.5		31.6	
1 week	28.9	28.7	29.2 29.2	32.2	31.7	31.7	30.4	30.2 30.4	33.5	33.2	33.2 33.2	33.3 33.2		31.4	49-58
2 weeks	28.7	28.5	28.7 28.8	31.8	31.6	31.5	30.2	30.5 30.1	33.3	32.9	33.1 33.5	33.2 33.5		31.2	32-55
4 weeks	28.3	28.5	28.7 28.7	31.4	31.2	31.0	29.9	29.8 29.9	32.9	32.7	33.1 33.0	33.0 33.1		30.9	40-76
6 weeks	27.8	28.1	28.3 28.1	31.0	30.6	30.5 30.6	29.5	29.6 29.5	32.5	32.2	32.4 32.4	32.4		30.4	41-78
8 weeks	26.8	27.6	27.5 27.4	30.5	30.1	30.0 30.1	29.1	29.3 29.0	32.0	31.4	31.6 31.5	31.9 31.8		29.8	34-70
3 months	26.0	27.0	26.6 26.8	29.7	29.5	29.5	28.4	29.0 28.1	31.5	30.8	31.0 30.8	30.6 30.7		29.1	51-80
4 months	25.6	26.3	25.7 25.6	28.9	28.8	28.8	27.5	26.8 26.5	30.9	30.0	30.5 30.3	29.9 29.7		28.3	50-93
5 months	24.6	25.0	24.3 24.1	28.0	27.7	27.6	26.5	25.4 25.1	30.2	28.1 27.6	29.3 29.5	28.8		27.1	72-92
6 months	23.0	23.9	23.2 23.3	26.9	26.2	26.0	25.6	24.2 24.0	29.2	26.5	28.7 28.6	27.6 27.7		25.9	72-90
7 months	21.8	22.8	22.3 22.1	25.7	25.0	23.0 22.4	24.0	23.0 23.2	29.1	25.4	27.8 28.0	26.3 26.2		24.6	68-90
8 months	20.0 19.3	21.7	20.6	24.4	23.9	21.3	21.9	21.9 22.6	27.0	24.2	26.5 26.3	24.9 24.7		23.2	50-88
9 months	18.4	20.2	19.3 19.1	23.0	22.1	20.0	20.4	21.0 20.7	25.8	23.2	25.0 24.8	23.5 23.5		21.8	40-80
10 months	17.6	19.0	17.8 18.0	21.9	21.3	18.8	18.8	19.8 19.7	24.6	22.0	23.4 23.0	22.0 21.8		20.6	38-60
11 months	17.0	18.5	16.8 16.5	20.6	20.7	18.0 18.1	18.4	19.0 19.4	23.7	21.9	22.1 22.3	21.5 21.4		19.9	38-66
12 months	17.0 18.0	18.0	15.7 15.9	21.7 20.4 20.2	20.4	17.0 16.8	18.0	19.4 19.0 18.6	22.9	23.2 21.5 21.7	21.2 21.3	20.7 21.0		19.5	34-60

Complete analyses of each sample, before and after storage, are reported in Table 4.

TABLE 4.—*Composition of chlorinated lime before and after storage*

SUMMER SERIES (PERIOD OF STORAGE 15 MONTHS)

Container	Composition									
	Calcium oxide (CaO)	Magnesium oxide (MgO)	Iron and aluminum oxides (R_2O_3)	Available chlorine	Chloride chlorine	Chlorate chlorine	Carbon dioxide (CO_2)	Silica (SiO_2)	Oxygen correction [1]	Water, etc. (by difference)
Packer A:	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12-ounce—										
Before	47.61	1.24	0.24	26.45	4.00	0.30	0.70	1.10	−0.56	18.92
After	47.28	1.40	.55	15.90	14.25	.25	1.26	1.05	−2.92	20.98
12-ounce—										
Before	46.77	1.21	.27	26.45	4.00	.20	.65	1.20	−.68	19.93
After	46.94	1.36	.59	16.30	13.90	.20	1.12	1.10	−2.90	21.39
10-pound—										
Before	46.88	1.20	.23	27.35	6.17	.40	.77	1.16	−.94	16.78
After	47.50	1.27	.48	18.20	13.90	.10	1.59	1.00	−3.02	18.98
Packer B:										
12-ounce—										
Before	46.56	1.20	.28	27.85	3.00	.20	.63	1.31	−.45	19.42
After	45.92	.96	1.43	15.00	15.40	.40	1.27	1.06	−3.02	21.58
12-ounce—										
Before	47.29	1.08	.28	27.40	3.10	.25	.51	1.32	−.42	19.19
After	46.04	1.08	1.49	16.00	14.50	.30	1.14	1.06	−2.93	21.32
10-pound—										
Before	47.08	1.20	.30	27.20	2.45	.20	.70	1.29	−.33	19.91
After	50.68	1.55	1.24	15.90	15.40	.20	1.26	1.05	−3.24	15.96
Packer C:										
12-ounce—										
Before	44.32	1.06	.28	28.50	6.90	1.10	.28	1.21	−.32	16.67
After	43.56	1.13	.50	1.90	28.40	.90	1.50	.84	−5.38	26.65
12-ounce—										
Before	44.26	1.09	.30	28.60	6.90	.90	.28	1.20	−.54	17.01
After	43.67	1.13	.50	4.80	25.30	1.00	2.23	.80	−4.57	25.14
5-pound—										
Before	44.47	1.24	.29	28.55	7.05	.60	.27	1.16	−.91	17.28
After	44.18	1.16	.53	9.40	22.90	.70	1.59	.92	−4.37	22.99
Packer D:										
5-ounce—										
Before	46.25	1.72	1.05	29.30	4.47	.28	.70	1.25	−.69	15.67
After	44.01	1.13	1.02	5.95	25.15	.30	1.22	1.26	−5.32	25.28
12-ounce—										
Before	46.09	1.75	1.23	29.15	4.70	.35	.67	1.28	−.66	15.44
After	44.75	1.23	.98	7.40	24.30	.30	1.25	1.25	−5.13	23.67
5-pound—										
Before	45.83	1.84	1.15	28.85	4.75	.30	.69	1.30	−.73	16.02
After	43.11	1.06	.91	6.60	24.60	.30	1.26	.92	−5.20	26.44
10-pound—										
Before	45.94	1.74	1.26	29.20	4.80	.40	.70	1.20	−.63	15.39
After	43.44	1.12	1.33	19.80	12.30	.40	1.04	1.05	−2.32	21.84
Packer E:										
10-pound—										
Before	45.47	.59	.35	34.10	3.20	.30	.63	.65	−.38	15.09
After	46.16	.81	1.00	21.10	16.30	.30	.80	1.65	−3.33	15.21

WINTER SERIES (PERIOD OF STORAGE 12 MONTHS)

Packer A:										
12-ounce—										
Before	46.91	1.67	0.42	29.20	2.90	0.30	0.62	0.90	−0.32	17.40
After	47.39	1.40	.58	15.90	15.80	.20	1.28	1.16	−3.33	19.62
12-ounce—										
Before	46.91	1.71	.45	29.15	2.75	.35	.61	.82	−.47	17.72
After	46.94	1.36	.60	15.70	16.15	.15	1.39	1.08	−3.47	20.10
5-pound—										
Before	47.26	1.77	.35	28.70	3.80	.30	.51	.74	−.52	17.09
After	47.61	1.27	.50	18.00	13.90	.20	1.21	1.00	−2.90	19.21
10-pound—										
Before	46.91	1.87	.62	28.90	3.00	.30	.54	.84	−.34	17.36
After	47.95	1.33	.58	18.00	14.20	.30	1.21	1.10	−2.86	18.19

[1] Oxygen correction is the algebraic sum of the positive oxygen equivalent of chlorate chlorine and the negative oxygen of the chloride chlorine.

TABLE 4.—*Composition of chlorinated lime before and after storage*—Continued

WINTER SERIES (PERIOD OF STORAGE 12 MONTHS)—Continued

Container	Composition									
	Calcium oxide (CaO)	Magnesium oxide (MgO)	Iron and aluminum oxides (R_2O_3)	Available chlorine	Chloride chlorine	Chlorate chlorine	Carbon dioxide (CO_2)	Silica (SiO_2)	Oxygen correction	Water, etc. (by difference)
Packer B:	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
8-ounce—										
Before	45.26	1.09	0.41	31.70	0.55	0.15	0.86	0.84	+0.05	19.09
After	45.58	1.16	.58	17.00	14.35	.30	1.43	1.10	−2.89	21.39
8-ounce—										
Before	45.02	1.11	.36	31.70	.60	.20	.83	.84	+.09	19.25
After	45.25	1.14	.60	16.80	14.40	.40	1.33	1.00	−2.79	21.87
5-pound—										
Before	45.84	1.20	.34	31.70	.60	.25	.54	.74	+.15	18.64
After	45.81	1.14	.53	20.40	12.00	.40	1.21	.98	−2.25	19.78
10-pound—										
Before	45.96	1.20	.38	32.00	.15	.15	.60	.74	+.14	18.68
After	46.04	1.08	.50	20.46	11.20	.35	1.19	.95	−2.13	20.36
Packer C:										
12-ounce—										
Before	47.62	2.26	.51	30.15	2.65	.25	.83	.85	−.31	15.19
After	47.27	2.19	.51	19.40	12.10	.40	1.39	.90	−2.27	18.11
12-ounce—										
Before	47.85	2.27	.48	30.40	2.50	.20	.86	.85	−.34	14.93
After	47.50	2.11	.48	18.60	13.30	.40	1.59	.88	−2.54	17.68
5-pound—										
Before	46.97	2.17	.65	30.40	2.80	.45	.69	.72	−.12	15.27
After	47.16	2.03	.54	18.00	14.00	.30	1.21	.91	−2.81	18.66
Packer D:										
5-ounce—										
Before	47.50	1.42	.63	33.25	1.10	.15	.61	.89	−.08	14.53
After	47.50	1.30	.80	20.70	13.60	.35	1.14	.95	−2.67	16.33
5-ounce—										
Before	47.26	1.42	.50	33.15	1.15	.20	.61	.98	−.03	14.76
After	47.33	1.35	.84	21.00	13.20	.60	1.17	1.00	−2.30	15.81
12-ounce—										
Before	46.41	1.52	.59	33.15	.95	.05	.61	.89	−.16	15.99
After	47.11	1.44	.83	21.30	12.90	.35	1.09	.96	−2.51	16.53
5-pound—										
Before	45.96	1.46	.51	33.20	1.20	.20	.53	.98	−.05	16.01
After	46.82	1.30	.72	21.50	12.90	.30	1.02	.99	−2.57	17.02
10-pound—										
Before	46.44	1.52	.50	33.50	1.35	.15	.62	1.06	−.14	15.00
After	46.94	1.16	.69	22.90	12.90	.50	.99	1.02	−2.34	15.24

The analytical results obtained on two samples of chlorinated lime imported from England, one subsampled into 5-ounce waxed cardboard containers with metal tops and the other into glass bottles with paraffined tops and kept on a dark shelf, are given in Table 5.

TABLE 5.—*Loss of available chlorine in high-test chlorinated lime (two imported samples) packed in fiber containers and in glass jars*

Storage period	Available chlorine in chlorinated lime packed in—		Storage period	Available chlorine in chlorinated lime packed in—	
	Fiber containers [1]	Glass jars [2]		Fiber containers [1]	Glass jars [2]
	Per cent	*Per cent*		*Per cent*	*Per cent*
When packed	35.6	34.6	7 months	26.2	28.8
1 month	35.1	34.0	8 months	24.4	27.5
2 months	34.5	33.0	9 months	20.5	26.4
3 months	33.8	32.2	10 months	18.7	25.2
4 months	32.4	31.8	11 months	----------	24.0
5 months	30.9	31.2	12 months	----------	22.4
6 months	29.9	30.1	Average monthly loss	1.69	1.02

[1] Packed Feb. 15, 1923.

[2] Packed Dec. 22, 1922.

The effect of the container on a 10-pound sample of bleaching powder (32.5 per cent available chlorine and 37.86 per cent total chlorine) received February 5, subsampled on April 8, packed, and stored in the dark for one and three months, is shown in Table 6.

TABLE 6.—*Effect of container on available chlorine lost during storage*

Condition of sample	Available chlorine in—	
	Sample stored in glass bottles with wax-covered corks	Sample stored in waxed cardboard cans with metal ends
	Per cent	*Per cent*
As received (Feb. 5)	32.5	32.5
When subsampled (Apr. 8)	32.5	32.5
Stored 1 month	31.0	29.3
Stored 3 months	28.5	22.9

The effect of light on a sample of chlorinated lime received February 17 and containing on May 23, when it was subdivided into three series of samples, 29.3 per cent of available chlorine and 36.5 per cent of total chlorine, is shown in Table 7.

TABLE 7.—*Effect of light on deterioration of chlorinated lime stored for 6½ months*

Storage period	Available chlorine in—		
	Samples in glass bottles with waxed corks		Samples in pasteboard 12-ounce cans, stored in dark
	Stored in dark	Stored in light	
	Per cent	*Per cent*	*Per cent*
When packed (May 23)	29.3	29.3	29.3
1½ months	25.9	24.1	24.6
2½ months	23.6	21.5	21.6
3½ months	22.8	20.0	18.9
4½ months	22.1	17.7	15.6
5½ months	21.4	16.6	14.4
6½ months	20.4	15.4	12.5
Average monthly loss	1.37	2.14	2.58

The deterioration of chlorinated lime packed in 8-ounce glass bottles fitted with cork stoppers and paraffined and exposed to diffused light for a year, as compared with the deterioration of the same samples when stored in a dark closet, is shown in Table 8.

TABLE 8.—*Effect of light on deterioration of chlorinated lime stored for 12 months*

SUMMER SERIES [1]

Storage period	Packer A		Packer B		Packer D		Packer E		Average	
	Dark	Light	Dark	Light	Dark	Light	Dark	Light	Dark	Light
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
When packed	26.1	26.1	27.2	27.2	29.0	29.0	34.1	34.1	29.1	29.1
1 month	25.4	25.4	26.0	26.0	27.2	27.4	32.8	32.9	27.8	27.9
2 months	25.1	25.0	25.6	25.6	27.0	27.1	31.2	31.5	27.2	27.3
3 months	24.7	24.5	25.3	25.3	26.7	26.5	31.7	31.2	27.1	26.9
4 months	24.1	23.9	25.0	24.9	26.3	25.9	31.5	30.8	26.7	26.4
5 months	23.4	22.8	24.6	24.3	26.0	25.5	30.7	29.8	26.2	25.6
6 months	22.7	21.7	23.8	23.2	25.0	24.3	29.7	28.7	25.3	24.5
7 months	21.8	20.7	23.0	22.2	24.1	23.0	28.6	27.8	24.4	23.4
8 months	21.3	20.1	22.5	21.1	[3] 22.0	21.8	27.5	27.3	23.3	22.6
9 months	20.4	19.4	20.7	20.7	21.8	20.7	26.5	25.9	22.4	21.7
10 months	19.4	18.8	19.7	19.8	20.6	19.4	25.4	24.5	21.3	20.6
11 months	18.2	17.9	19.3	18.4	19.6	18.4	24.3	23.3	20.4	19.5
12 months	17.2	16.8	17.9	16.8	18.8	17.5	23.2	22.4	19.3	18.4
Average total loss									9.8	10.7
Average monthly loss									.82	.89

WINTER SERIES [2]

Storage period	Packer A		Packer B		Packer C		Packer D		Average	
	Dark	Light	Dark	Light	Dark	Light	Dark	Light	Dark	Light
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
When packed	29.6	29.6	32.4	32.4	30.5	30.5	33.5	33.5	31.5	31.5
1 month	28.7	28.7	31.8	31.8	30.1	30.0	32.6	32.5	30.8	30.8
2 months	28.3	28.2	31.5	31.3	29.2	28.8	32.0	31.8	30.3	30.0
3 months	27.6	27.4	30.8	30.7	28.5	28.4	31.2	31.0	29.5	29.4
4 months	26.3	25.8	29.9	29.8	27.2	27.2	30.8	30.5	28.6	28.3
5 months	25.8	25.3	29.2	29.0	26.8	26.7	30.1	30.0	28.0	27.7
6 months	25.3	24.7	28.4	28.1	25.9	25.7	29.2	29.0	27.2	26.9
7 months	24.5	24.1	27.4	27.0	25.3	25.1	28.5	28.2	26.4	26.1
8 months	24.1	23.5	26.7	26.2	24.5	24.2	28.0	27.5	25.8	25.4
9 months	23.6	22.4	26.0	25.1	24.0	23.7	27.6	27.0	25.3	24.6
10 months	23.0	21.5	25.0	24.0	23.2	22.8	27.1	26.3	24.6	23.7
11 months	22.5	20.4	24.3	23.2	22.4	22.0	26.4	25.5	23.9	22.8
12 months	20.9	20.0	23.9	22.7	21.9	21.4	25.8	24.9	23.1	22.3
Average total loss									8.4	9.2
Average monthly loss									.70	.77

[1] Packed Sept. 30, 1922. [2] Packed Feb. 15, 1923. [3] Slight explosion occurred when bottle was opened.

The average total loss and average monthly loss from containers of various sizes and types for the combined summer-packed and winter-packed material are shown in Table 9.

TABLE 9.—*Effect of size and type of container on deterioration of chlorinated lime*

Sample	Average total loss after—		Average monthly loss after—	
	6 months	12 months	6 months	12 months
Summer and winter-packed material from packers A, B, C, D, and E, stored in—	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Glass bottles in the dark	4.1	9.1	0.68	0.76
5 and 10 pound metal containers	4.7	10.6	.78	.88
Fiber containers	5.3	13.1	.88	1.09
Winter-packed material from packers A, B, C, D, and E and summer-packed material from packers A, B, and E, stored in—				
5 and 10 pound metal containers	4.7	10.5	.68	.88
Fiber containers	5.3	11.7	.88	.98

DISCUSSION OF RESULTS

The deterioration of the summer-packed material, as measured by the loss of available chlorine, was regular for the first nine months. (Table 3 and figs. 2 and 3.) After this time samples from packers C and D deteriorated more rapidly than those from packers A, B, and E. The material put up by packers C and D came from different manufacturers, who also made material for packers A and E. The excessive deterioration is therefore ascribed to the type of container in which the samples were packed rather than to the method of manufacture. At the end of the experiment the samples from packers C and D contained more water than those from packers A, B, and E

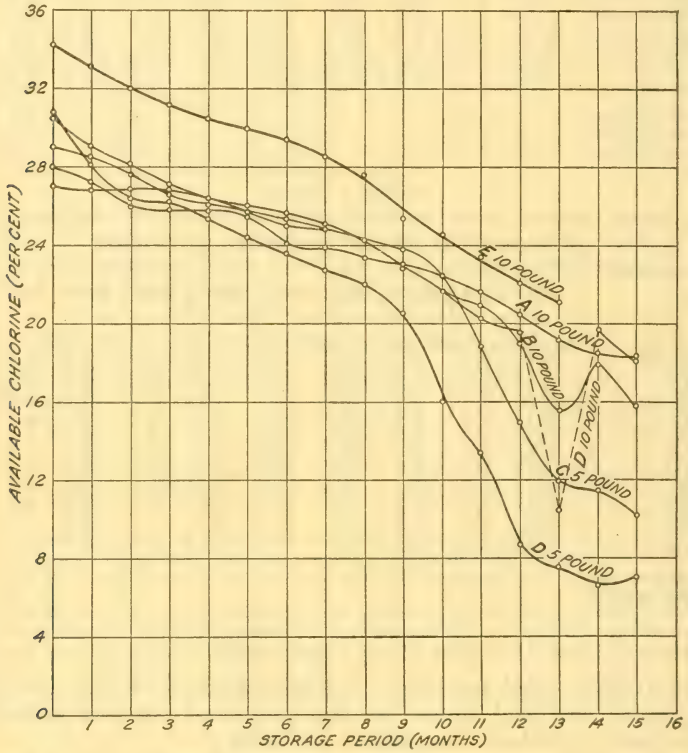

FIG. 2.—Loss of available chlorine in summer-packed chlorinated lime stored in 5 and 10 pound packages for 16 months

indicating that the containers were not tight. The subsamples from packers C and D kept in glass bottles deteriorated at about the same rate as those from packers A, B, and E. Thus it is evident that the difference in the behavior of the various series of samples was due to the containers in which they were packed.

During the experiment the paraffined fiber containers of all sizes used by packer C and those of the 5-pound size used by packer D became brittle after 5 or 6 months. The 12-ounce containers of packer C became noticeably brittle after storage for 3 months. The other containers of packers C and D showed evidence of deterioration after standing for 8 to 9 months.

The winter-packed samples showed a regular rate of deterioration throughout the experiment. (Table 3.) The rate of loss of available chlorine from them was more uniform than that from the summer-packed material. (Fig. 4.)

EFFECT OF SIZE AND TYPE OF CONTAINER

With any one type of container the size appeared to have little effect. The chlorinated lime packed in 5-ounce, 12-ounce, and 5-pound fiber containers seemed to deteriorate at about the same rate. (Table 3.)

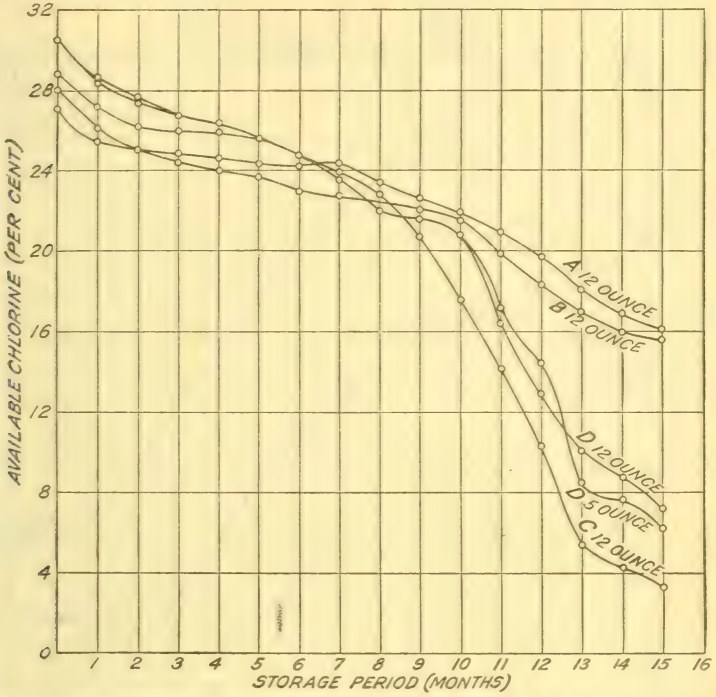

FIG. 3.—Loss of available chlorine in summer-packed chlorinated lime stored in 5 and 12 ounce packages for 16 months

The larger (5 and 10 pound) metal containers were slightly superior to the others from the standpoint of preventing decrease in available chlorine content. (Table 9.) The best container is evidently the tightly-stoppered glass bottle. This is to be expected from consideration of the fact that the deterioration rate seems to be hastened by the absorption of moisture.

EFFECT OF LIGHT

In one series of experiments, in which a sample of chlorinated lime was stored for 6½ months, the material that was exposed to the light deteriorated much more rapidly than that which was kept in the dark. (Table 7.) In another series, however, in which eight samples were stored for 12 months, the material stored in the light deteriorated only slightly more than that stored in the dark. (Table 8.) In the first series the average monthly deterioration was 1.37 per cent for

the material stored in the dark and 2.14 per cent for that stored in diffused light. In the second series the average monthly loss was 0.76 per cent for the material stored in the dark and 0.83 per cent for that stored in diffused light. Light rays therefore appear to accelerate slightly the decomposition of chlorinated lime.

EFFECT OF SEASONAL VARIATION

During the first nine months of storage the average loss of available chlorine was 0.78 per cent per month from samples manufactured and packed in the summer and 1.09 per cent from those manufactured and

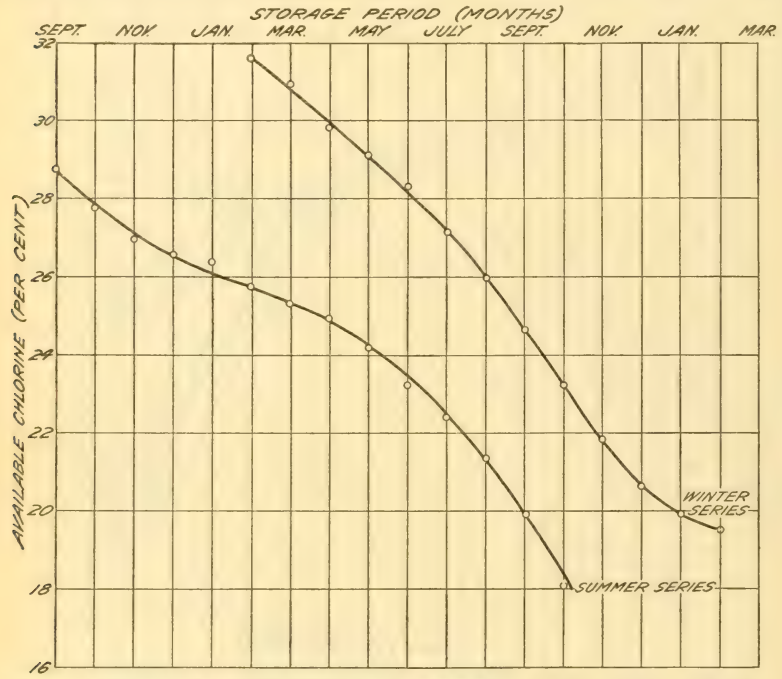

FIG. 4.—Average loss of available chlorine in summer-packed and winter-packed chlorinated lime stored for 16 months

packed in the winter. The fact that the winter-packed material was subjected to summer temperature for a longer period, beginning about three months after storage, probably accounts for its greater deterioration. After nine months summer samples from packers C and D showed a rapidly accelerated rate of loss of available chlorine. As this increase occurred only after nine months, it can scarcely be ascribed to the season of manufacturing or packing the chlorinated lime. Omitting the values for these two samples, the average monthly loss for the 12-month period is 0.89 per cent for the summer samples and 1.01 per cent for the winter samples.

A plot of the monthly rate of loss of each series of samples according to calendar months (fig. 5) shows a distinct seasonal variation. The curves for the two series are practically parallel and show that the

rate of loss of available chlorine is higher during the summer months and lower during the cooler fall and winter months, a fact that had previously been recognized. The values for samples from packers C and D, which were considered abnormal, were not used in plotting the curve for the summer samples.

EFFECT OF METHOD OF PACKING

Samples from packer C were mixed and packed entirely by hand; those from all the others were packed by machinery. Packer B used a machine with a foot-tread control to feed the material into the container. The other packers used automatic feed machines.

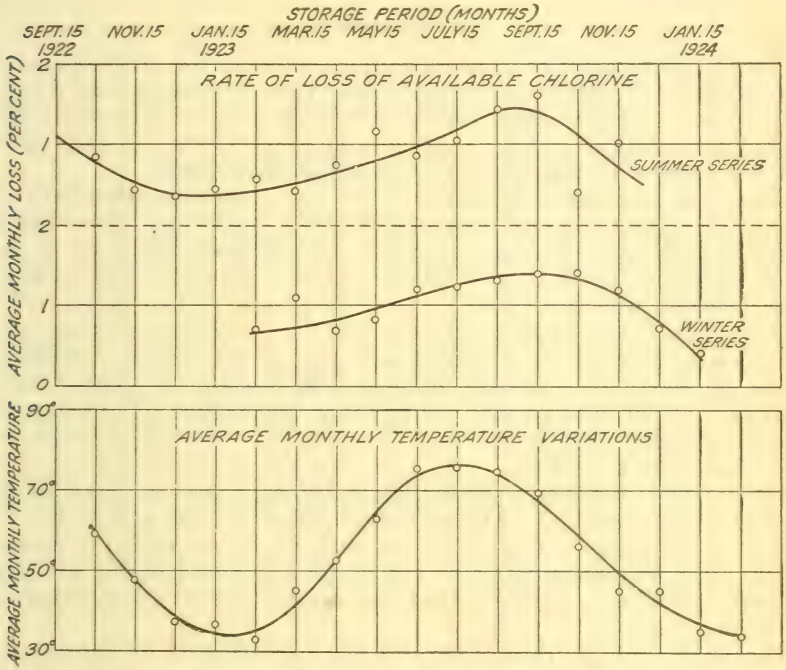

FIG. 5.—Rate of loss of available chlorine from winter-packed and summer-packed chlorinated lime stored at varying temperatures.

When the winter-packed samples were prepared the air was dry and the material packed by hand was as stable as that packed by machinery. When the summer-packed samples were put up by packer C the humidity was high and the material was spread on trays open to the atmosphere for about two hours during the packing, which should have given a good chance for the absorption of moisture and carbon dioxide from the air. This material, although rather lumpy when first opened, contained no more water or carbon dioxide than that packed by machinery. For the first nine months it kept as well as the others, but at the end of this time it rapidly lost available chlorine. As the material from packer D showed a similar loss, however, this variation can not be attributed to the method of packing.

EFFECT OF DETERIORATION ON CHEMICAL COMPOSITION

The chemical composition of the samples from the various packers (Table 4) was uniform. No connection between the rate of decomposition and the composition of the samples is indicated.

The determination of available chlorine and chloride chlorine at the beginning and at the end of the storage period showed that nearly all of the available chlorine was converted into chloride and that very little was lost by volatilization. There were evidences of a small loss in most cases, however, the losses being most pronounced in summer-packed samples from packers C and D. These samples showed the greatest losses of available chlorine, as well as the greatest losses in actual chlorine.

SUMMARY

The available chlorine in most samples of bleaching powder packed in containers of the usual commercial types decreased fairly regularly during storage. Most of the available chlorine lost was transformed into chloride chlorine, the loss in total chlorine in most cases and the change in chlorate chlorine being very slight.

There was no marked difference in the rate of deterioration between the samples manufactured and packed in the winter and those manufactured and packed in the summer. There was, however, a seasonal variation, the loss of available chlorine being greater in warm than in cold weather.

In general, the keeping qualities of the bleaching powder were not greatly affected by the type and size of commercial container. There appeared to be a small difference in favor of the larger metal containers over the other commercial packages. The material stored in tightly stoppered glass bottles deteriorated slightly less than that stored in cans.

Two of the samples of bleaching powder packed in the summer showed about the same rate of deterioration as the others for the first nine months. After that the rate of deterioration increased rapidly and was accompanied by a decided increase in the moisture content. This may indicate that the absorption of water catalyzes the change.

The results on the rate of deterioration as affected by the method of packing did not lead to any definite conclusions. The hand-packed material put up in the summer showed a high rate of loss after about 10 months. As one of the machine-packed samples also showed a high rate of loss during the same time, however, this can not definitely be attributed to the method of packing.

The visible rays of light seem to have a slight accelerating effect on the loss of available chlorine.

The average loss in available chlorine for all samples from the different sources, packed and stored in various ways, was 1.08 per cent per month for the period covered, 12 to 15 months, the average for the hottest months (May 15 to September 15) being 1.44 per cent per month and for the coldest months (November 15 to March 15) 0.61 per cent per month.

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